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(54) GLASS COMPOSITION

(71) We, OWENS-CORNING FIBERGLAS CORPORATION, a corporation organized and existing under the laws of the State of Delaware, United States of America, of Toledo, Ohio, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to glass compositions, and more particularly, compositions free of borates and fluorides which lend themselves well to the manufacture of textile fibers. The present compositions are free of alkali ingredients and replace a composition known as "E" glass and similar glasses used extensively by manufacturers of glass fibers, especially textile fibers.

Extensive development programs have been conducted for many years to improve textile glass fibers; however, except for specialty glasses, better glass compositions have not been found until now.

The liquidus-viscosity relationship of glass compositions to be commercially fiberized is one of the critical factors to be considered by fiber producers. The requirements for the production of continuous fibers (textile) are more stringent than that for discontinuous fibers (wool). Not all glasses that can be fiberized by some presently available process can be formed into continuous fibers commercially. Until the present invention, the formation of commercially acceptable continuous fibers at commercial speeds could be accomplished only with E glass (a boro silicate glass used in manufacturing textile fibers) which has been used extensively for over 30 years, and 621 glass (see U.S. Patent Specification No. 2,571,074) which has been used for a somewhat shorter time.

Present-day uses require higher temperature resistance, greater strength, improved abrasion resistance, and increased chemical durability over those properties obtained with E and 621 glass. Acid-resistance greater

than that provided by formerly available commercial textile fibers is also now needed. The constantly greater need for finer fibers requires consequently greater precision in control of the attenuating operation, viscosity of the glass, and homogeneity of the glass.

The glass preferably must be formed from low cost, natural-occurring materials that can be melted with relatively low evolution of volatiles that pollute the atmosphere. Large-scale production by industrial plants throughout the nation has resulted in fear of air pollution with attendant legislation directed toward alleviation of air pollution problems.

It is an object of this invention to provide an improved, high strength, high modulus, commercially formable glass composition in fibrous form suitable for all textile fiber purposes.

It is an object to provide glass compositions that fulfil the stringent requirements with respect to liquidus-viscosity relationship for commercial production of continuous glass fibers.

It is an object to improve the durability of fibers and particularly durability with respect to acid and having higher strengths and higher modulus of elasticity than those formerly attainable with commercial glass fibers in continuous textile forms.

It is an object to provide a glass composition which can be derived from a simple combination of natural occurring minerals and preferably including no rare or expensive ingredients and free of borates, fluorides and other such volatile and/or condensable materials.

The new compositions disclosed herein include silica, alumina, magnesia and calcia in the proportions expressed in the examples which follow. These compositions are preferably free of alkali ingredients, B₂O₃, and fluorides except for possible traces not purposely added. They are capable of being fiberized into continuous fibers

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by commercial processes with no difficulty.

Silica fibers are known to exhibit good resistance to high temperature. They are however, difficult to fiberize. Silica, alumina, calcia and/or magnesia are combined in the indicated proportions to provide compositions having a desirable viscosity-temperature relationship and favorable devitrification rate for continuous fiber production.

It has been found that these oxides can be combined in the following proportions.

SiO ₂ ...	50—68% by weight
Al ₂ O ₃ ...	12—32
CaO ...	0—28
MgO ...	0—23

More particularly, in accordance with the invention there are provided glass fibers comprising from 50—68% silica, from 12—32% alumina, the remainder being calcia and/or magnesia, the total of silica and alumina being from 80—86%, all per cents being by weight.

These compositions provide high temperature resistant, high strength fibers. It has been found that when SiO₂ is 65%, Al₂O₃ is preferably equal to or less than 16%. When the SiO₂ is 60%, Al₂O₃ is equal to or more than 20%. When the SiO₂ is 55%, Al₂O₃ is equal to or more than 25%. Although both calcia and magnesia are shown as low as zero, both these oxides are not reduced to zero at one time, but rather at least 14% of calcia and/or magnesia alone or combined is present as a minimum.

EXAMPLE 1

SiO ₂ ...	60 parts by weight
Al ₂ O ₃ ...	20
CaO ...	12
MgO ...	8
Liquidus ...	2405°F.

This glass composition was melted from batch and fiberized by passing the glass through a plurality of orifices in a tip section of a feeder maintained at a temperature of about 2640°F. to produce K18's textile fibers. (K18 indicates a diameter of 0.00050 to 0.000549 and 1800 yards per pound) K18's are produced from a feeder having about 800 orifices in a single feeder. The fibers are from 50—55 × 10⁻³ inch in diameter. The tensile strength of virgin fibers was 600,000 psi, this being an average, and the modulus of elasticity was 12.4 × 10⁶ psi as determined by measuring elongation of the fiber under load.

EXAMPLE 2

SiO ₂ ...	65 parts by weight
Al ₂ O ₃ ...	15
CaO ...	10
MgO ...	10

The liquidus of this glass is 2430°F. Fibers were formed by flowing molten glass through a feeder maintained at a temperature of 2620°F.

EXAMPLE 3

SiO ₂ ...	50 parts by weight
Al ₂ O ₃ ...	30
CaO ...	10
MgO ...	10

The liquidus is 2517°. Glass Fibers were formed from a melt by passing the glass through a feeder to form streams of molten glass that are attenuated into glass fibers.

EXAMPLE 4

SiO ₂ ...	60 parts by weight
Al ₂ O ₃ ...	25
CaO ...	10
MgO ...	5

The liquidus of this composition is 2480°F. Glass fibers were produced by flowing streams of glass through a feeder at a temperature of 2620°F. and these streams then attenuated into fibers by wrapping the fiber onto a collet.

EXAMPLE 5

SiO ₂ ...	55 parts by weight
Al ₂ O ₃ ...	30
CaO ...	5
MgO ...	10

Fibers were formed in the same manner as above.

The glass fibers are produced from glass compositions formulated to be essentially free of alkali ingredients, fluorides, boron oxides and other such volatile and/or condensable materials. Traces of these impurities (fractional per cents) may be present but are not purposely added although they may be detected by present analytical techniques.

The above compositions are fiberized in accordance with present commercial practice involving the melting of the batch ingredients in a furnace and flowing this melt through feeders in the form of multiple streams of glass that are attenuated into individual fibers. Two hundred fibers, eight hundred fibers, or up to one thousand or more, are attenuated from a single feeder. These fibers are gathered together into a strand which is wound upon a collet winder such as that shown in U.S. Patent Specification No. 2,391,370. Fibers are formed into strands and the strands are combined then into yarns by conventional twisting and plying techniques. The fibers of Example 1 have been tested extensively. These fibers are superior to conventional E glass fibers as shown in the following table:

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TABLE 1

	"E" Glass	Example 1	% Improvement
Tensile Strength, psi	530,000	600,000	13%
Modulus of Elasticity, psi	10.5×10^6	12.4×10^6	18%
5 Fiber Chemical Durability (wt. loss) — 7 days at 90°C.			
Water	1.1	0.44	60%
H ₂ SO ₄ (50%)	8.9	0.54	94%
10 Rod Flexural Strength psi $\times 10^3$			
Dry	175.8	181.8	3.4%
Wet	134.8	150.6	11.7%
% Retention	76.7	82.8	8.0%

This table shows tensile strength, modulus, chemical durability and rod flexural strength for E glass and the glass of Example 1.

In arriving at the figures for chemical durability, fibers of E glass and fibers of Example 1 were subjected to water and to 50% H₂SO₄ by immersing them for 7 days at 90°C. and weight loss was measured with 60% and 94% improvement respectively over E glass. The rod flexural strengths were derived from testing samples of roving reinforced polyester rods. Fibers used to make these test rods were treated with 806 size which comprises polyvinyl acetate, vinyl trichlorosilane and lubricants in a water system. Both E glass fibers in a roving form and the Example 1 glass fibers in roving form were made into rods after they were treated at forming with the above size composition. A direct comparison of flexural strength is shown in Table I. Tensile strength is 13% improved, modulus of elasticity 18% improved, and retention of rod flexural strength is improved 11.7% when specimens are immersed 4 hours in boiling distilled water.

The glasses of this invention are unusual in that they are fiberizable even though they lie close to areas of glass compositions having viscosity-temperature relations unfavorable for continuous fiber production; that is, they are commercially fiberizable even though other compositions in the silica, alumina, calcia, magnesia area are not readily formed into continuous textile fibers by commercial methods. The glass may be produced from potter's flint, clay, and dolomite when Example 1 composition is desired. No expensive B₂O₃ is required. For this reason, such glasses are economical and yet provide improved properties not achievable with former glass compositions

commercially utilized in the continuous fiber process.

Strands and yarns or fabrics produced from these continuous fibers can be heat cleaned at higher temperatures than those conventionally used for cleaning E glass fabrics. The slump temperature of these fibers is 200°F higher than that of E glass fibers. Less fuzz and fly is produced while beaming these yarns prior to weaving or making a reinforced paper product. The compositions are especially suited for continuous fiber formation but they can be used for textile staple fibers.

WHAT WE CLAIM IS:—

1. Glass fibers comprising from 50—68% silica, from 12—32% alumina, the remainder being calcia and/or magnesia, the total of silica and alumina being from 80—86%, all per cents being by weight.
2. Glass fibers as claimed in claim 1 comprising 60% or more of silica.
3. Glass fibers as claimed in claim 1 or claim 2 comprising 20% or more of alumina.
4. Glass fibers as claimed in any preceding claim, the said remainder being alkaline earth oxides obtained from dolomite.
5. Glass fibers as claimed in claim 1 comprising by weight, 60% silica, 20% Al₂O₃, 12% CaO and 8% MgO.
6. Glass fibers as claimed in claim 1 comprising by weight, 65% silica, 15% alumina, 10% calcia, and 10% magnesia.
7. Glass fibers as claimed in claim 1 comprising by weight, 50% silica, 30% alumina, 10% calcia and 10% magnesia.
8. Glass fibers as claimed in claim 1 comprising substantially by weight, 60% silica, 25% alumina, 10% calcia and 5% magnesia.
9. Glass fibers as claimed in claim 1

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- comprising substantially by weight, 55% silica, 30% alumina, 5% calcia and 10% magnesia. ccribed herein with reference to the Examples.
- 5 10. A batch for producing continuous glass fibers having the composition set forth in claim 5 comprising potter's flint, clay and dolomite.
11. Glass fibers substantially as des-

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